#### ■ 儀器裝置

三電極系統



參考電極

工作電極

輔助電極



電位儀(Potentialstat/Galvanostat)

#### ■ 操作步驟



1. 研磨工作電極: 在兔毛研磨墊上放些許鋁粉、滴上少許 DI 水,進行電極表面拋光。

- 2. 參考電極:將前端保存液套筒移除。
- 將三支電極接線端藍色蓋子旋轉開、分別接上連接線。(工作電極和輔助電極連接線 為同類型)



 將三支電極分別連接至電位儀對應的插孔(工作=WORK、輔助=AUX、參考=REF),開 啟電位儀電源。



以 DI 水沖洗電極、拭鏡紙 按壓式 擦乾電極,再將三支電極插入玻璃杯孔中。
 <注意> 擦拭電極時請勿服貼式來回擦拭,避免電極表面刮傷。



將待測液倒入玻璃杯中,電極前端須完全浸入液面下且偵測端不可有氣泡(特別是工作電極)。若有氣泡請將電極拔出,用玻棒輕刮電極下方以去除氣泡。



7. 電腦開機,點選桌面上「VoltaMaster 4」進入測量畫面。



8. 點選 File→New Sequence,此時畫面出現「Logbook」視窗。





9. 點選 Settings→ Instrutment setup (圖 1) → Potentiostat 窗格選「VoltaLab PGP201」並 點擊「Test」(圖 2),此時 Instrument 顯示「PGP201」、Version 顯示「X0009」表電位 儀已連接(圖 3)→ 按下 OK 完成設定。

🚰 VoltaMaster 4 - [Untitled2 - Laboratory logbook ]				
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Settings	Settings
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(圖 2)	(圖 3)

10. 點選 Sequence → Sequence edition (圖 4) →點選 Voltammetry (圖 5) → 將「Pot. Cyclic

<u>Votalmmetry</u>」反白,點選「Add」加至右側 Sequence 中。

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Untitled1 - Laboratory logbook           Logbook         Initial data	Tools	Pulse Corrosul Battery Tutor
Operator: Date: Specification of the test: Working Electrode: Reference Electrode: Auxiliary Electrode: (圖 4)	Method           Open Circuit Potential           Pot. Cyclic Voltammetry           Pot. Tutorial CV           Pot. Interactive CV           Pot. Linear V           Gal. Cyclic Voltammetry	Add     Pot. Cyclic Voltammetry       Insert     Pot. Cyclic Voltammetry       Insert     Pot. Cyclic Voltammetry       Add     Pot. Cyclic Voltammetry       Insert     Pot. Cyclic Voltammetry

(圖 5)

11. 連擊圖 5 視窗中「<u>Pot. Cyclic Votalmmetry</u>」兩下(或點選 edit)進入參數設定畫面:

(1) Potential 0 : <u>650 mV</u>  $\sim$  Potential 1 : <u>650 mV</u>  $\sim$  Potential 2 : <u>-300 mV</u>

(2) Scan rate : <u>10 mV/sec</u> > Step : <u>6 mV</u> > Record a number of <u>3 cycles</u>

設定完成後,按下 OK 回到 logbook 畫面。

Pot. Cyclic Voltammet	ry			X
Potentiostatic ramp Potential 0 650 Potential 1 650 Potential 2 -300 Scan rate 10 Step duration 0.6 Step 6 D/A OUT initial 0 D/A OUT final 0 A/D IN Open circuit at end	mV mV mV mV/sec. ▼ sec. mV mV	Maximum current Minimum current Priority auto ranging Maximum Minimum range Filter Ohmic Drop Comp. R to compensate Record a number of	1 -1 Auto Auto Auto No 0 3 K	A A A Cancel
Potentials are measured versus REF. Linear voltammetry is run if Potential 2 = Potential 1. If Potential 0 = Last or Free or OCP, amplitudes are set versus Last, Free or OCP value. If Potential 0 = numerical value, potentials are set versus REF.				

12. 點選畫面綠色三角形(Start)、選取儲存位置、開始測量。(測量畫面右下方藍色 bar 全滿後表測量結束)

🚟 YoltaMaster 4 - Untitled1 - Laboratory logbook	
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Type: Normal 💌 X Potential 💌 Y1 Current 💌 Y2 No	<b>_</b>

13. 選擇 Processing (圖 6) → Peak analysis, 依序設定下列參數:

(1) Base line 分頁(圖 7-1):選「Manual」(圖 7-2),輸入基線取點範圍 Point 1 <u>316</u>與
 Point 2 <u>364 (</u>助教解說)(圖 7-3)

(2) Results 分頁(圖 7-4): 輸入取點數 From <u>316</u>與 To <u>474</u>(助教解說)(圖 7-5),按下「Draw」(圖 7-6)記錄還原電流值(圖 7-7)。

(3)自行計算氧化區段取點數,取得氧化電流值。

Edit Quare       Processing oolbac       Window       Help         SubAdd       SubAdd       SubAdd         Liver       Egtaction       V2 No         Curve       Results       Point cursor         Manual slope       Liver regression       East         It form method:       Tafel       Statem method:         Results       Point cursor       Monual slope         Liver regression       East       Statem method:         It Stem method:       Tafel       Statem method:         Results       Point cursor       Monual slope         Liver regression       East       Statem method:         Go Calometric discolution       Rescression       Point 2         Point 2       Statem method:       Statem method:         Rescression       Point 1       Statem method:         Curve       Rescression       Point 2         Statem method:       No       Statem method:         Rescression       Point 1       Statem method:         Rescression       Point 2       Statem method:         Rescression       Point 2       Statem method:         Rescression       Point 2       Statem 2       Pointo: <td< th=""><th>🚆 VoltaMaster 4 - [nu</th><th>iknow005]</th><th></th><th></th></td<>	🚆 VoltaMaster 4 - [nu	iknow005]		
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14. 测定結束後將三支電極以 DI 水沖洗、拭鏡紙按壓式擦乾收起(參考電極須套回 3M KCl 套筒中保存),電位儀及電腦關機。